

Remarks

Claims 1-15 are currently pending in the present application, and new claim 16 is added herein.

In the Action mailed June 17, 2004, the examiner rejected claims 1-4, 6-8, 13 and 14 under 35 U.S.C. section 102(b) as allegedly being anticipated by, or in the alternative under 103(a) as allegedly being obvious over U.S. Patent No. 4,450,087 or U.S. patent No. 3,903,006 to Elliott et al.

In response to the rejection applicants provide the following distinguishing remarks that are believed to place the present case in condition for allowance. Favorable reconsideration of all of the pending claims is respectfully requested.

Initially, regarding the restriction requirement, applicants have added new "product-by-process claim 16 that effectively links product claim 1 and process claim 5. In view of this amendment, applicants respectfully request that claim 5 be included with pending claims 1-4, 6-8, 13 and 14 for prosecution on the merits.

Concerning the Declaration Under 37 C.F.R. § 1.132, compound 020219HR of Askew et al. should be disregarded as comparative tests were not conducted and this compound as this compound was not deemed to be particularly close in structure to the compounds of the present invention.

It is applicants' opinion that compound 020222HR2 of Askew et al. is the compound most closely related to the products of the present invention, and it is equal to the ortho ester component of Askew ex. 56.

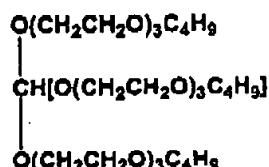
The compound 3xMPEG 350 has not been described in any working example of Askew, but is within the general formula of ortho esters belonging to the invention of Askew et al. It is the compound that applicants believe is the most structurally similar to the ortho esters of the present invention, having substituents that are the same as the hydrophilic substituents of the ortho esters of example 5 of

the present invention, except that the compounds of example 5 also contain a hydrophobic substituent (n-octyl).

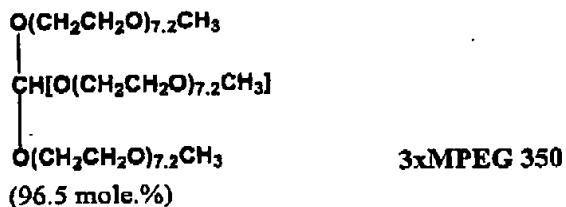
As requested by the examiner, applicants now provide the structural formulae of the compounds in question.

**Compound structures:**

According to U.S. Patent No. 4,450,087 to Askew et al.:



020222HR2 (*ortho ester part Askew ex.56*)



3xMPEG 350

(96.5 mole.%)

As can be clearly seen from the above structures, the hydrophobic parts (in these cases alkyl chains) are small as well as uniformly distributed over the entire molecule, thus not resulting in clearly separated hydrophilic and (sufficiently) hydrophobic domains. Such a separation is known to be a requirement for a compound to orient itself at the interface between a hydrophilic (e.g. water) and hydrophobic surface and thereby induce surfactancy.

The examiner is respectfully requested to note that all examples of Askew contain ortho esters that have three equal substituents, whereas the ortho esters according to the present invention never contain three equal substituents.

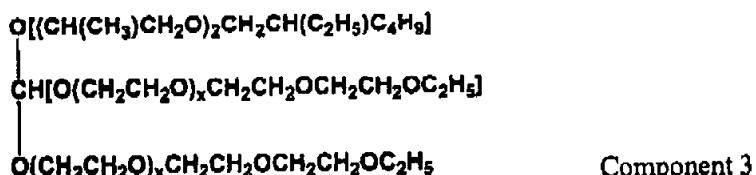
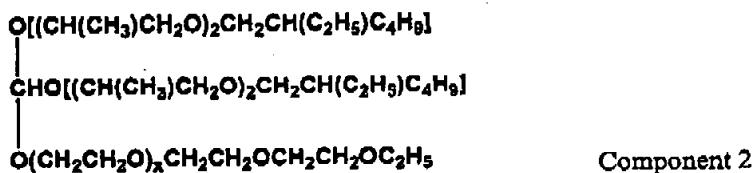
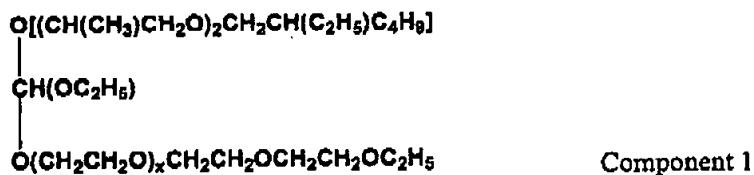
**Ortho esters according to the present Invention used in the comparison:**

Concerning the Declaration, compound GT 6220-02E of the invention should be disregarded as applicants have not performed any investigations on this

compound. The compounds according to the present invention that have been investigated are **GT 6220-05E**, **GT 6220-09E** and **GT 6220-12E**. These products have been made by first preparing the starting material for the hydrophilic part by ethoxylating ethyl diglycol (=  $C_2H_5OCH_2CH_2OCH_2CH_2OH$ ) with different amounts of ethylene oxide. For **GT 6220-05E** ethyl diglycol has been ethoxylated with 3 moles of ethylene oxide, and for **GT 6220-09E** and **GT 6220-12E** with 7 and 10 moles respectively. The hydrophobic part has been prepared by propoxylating 2-ethylhexanol with 2 moles of propylene oxide. Then the hydrophilic starting material, the hydrophobic starting material and triethyl orthoformate have been reacted together to obtain **GT 6220-05E**, **GT 6220-09E** and **GT 6220-12E**, respectively.

**Formulae representing the compounds according to the present invention:**

**GT 6220-05E** comprising the following three essential components (5 denotes the total amount of ethyleneoxy units per hydrophilic substituent: the symbol x is 3)



The same formulae apply for GT 6220-09E and GT 6220-12E where the symbol x is 7 and 10 respectively. Thus, as explained in the application, the products of the claimed invention are mixtures of components where the main surface chemical effect is given by the three components with

- a) one hydrophilic, one hydrophobic and one short alkyl chain (component 1),
- b) one hydrophilic and two hydrophobic chains (component 2) and
- c) two hydrophilic and one hydrophobic chain (component 3),

respectively. The hydrophilic chains are the ones containing ethyleneoxy groups that are ended with a short alkyl group (in this case ethyl). The ortho ester surfactants according to the present invention are displayed for GT 6220-05E as Component 1, 2 and 3 respectively as shown above. In contrast to the teaching of Askew, the present compounds also contain substituents of considerable hydrophobicity (2-ethyl-hexyl+2 propoxy). Further, the hydrophilic and hydrophobic entities are clearly located in different parts of the molecule. These differences thus are the basis for the unexpected results of the present invention. Additionally, the ortho ester link is connecting the hydrophilic and hydrophobic entities. Cleavage (hydrolysis) of this linkage thus results in a complete separation of these entities inevitably giving a loss of surfactancy. This is in contrast to the closest art of Askew,

where the hydrophilic and (too small) hydrophobic parts are still connected after such a link cleavage.

Concerning the actual data in applicants Declaration, applicants believe that a further detailed discussion herein is probably warranted.

### 1) Static Surface Tension

#### Method

Surface tensions were recorded using a KSV Sigma 70 tensiometer (KSV Instruments LDT, Helsinki, Finland) equipped with a du Noüy ring. Surfactant concentrations were 0.1 % (1 g/L) in pH 8 buffer solution(P-H TAMM Laboratorier AB, Uppsala, Sweden) in order to avoid any hydrolysis during the measurement. The buffer solution has a surface tension approximately the same as pure water (72 mN/m).

#### Results – Table 1

Product	Surface Tension (mN/m)
GT 6220-05E	29.7
020222HR2 (Comparison)	37.1
3xMPEG 350 (Comparison)	59.7

A basic feature of surfactants is the ability to lower the surface tension. It is generally recognized that good surfactants should be able to decrease the surface tension of water to less than *ca.* 30 mN/m. A compound with moderate efficiency gives results in the range of *ca.* 30-35 mN/m. As the data clearly demonstrate, the products of the present invention give rise to substantially lower surface tension values than the closest comparison products of Askew et al.

## 2) Dynamic Surface Tension

### Method

The dynamic surface tension for some of the products earlier described was in this example measured by using a Sensadyne PC500-L Surface Tensiometer (Chem-Dyne Research Corp., Mesa, Az., United States). 0.5 % (5g/L) surfactant solutions were used.

Dissolution in pH 8 buffer solution (P-H TAMM Laboratorier AB, Uppsala, Sweden) in order to avoid any hydrolysis during the measurement.

The surface tension was measured as a function of time. The solutions were continuously stirred except during the measurements. With the method used\*, the surface tension is measured by creation of nitrogen bubbles in the bulk phase. The process is dynamic, and the surface tension measured depends on the surface age of the bubble. A higher surface age normally means a lower surface tension until the creation time is so long that steady-state conditions are reached. The surface age at which this takes place is individual for each surfactant.

\* The method is described in e.g. U. Telpel and N. Aksel, *Tenside Surf. Det.* 37 (2000) 5, 297-308 and references herein.

## Results

**Table 2**

Product	Surface age (s)	Surface Tension (mN/m)	Surface age (s)	Surface Tension (mN/m)
<b>GT 6220-05E</b>	0.65	31.6	2.34	29.7
<b>GT 6220-09E</b>	0.69	31.0	2.46	29.4
<b>GT 6220-12E</b>	0.64	34.8	2.49	29.8
<b>020222HR2 (Comparison)</b>	0.62	35.7	2.89	36.4
<b>3xMPEG 350 (Comparison)</b>	0.67	63.5	2.70	63.2

As for the static surface tension, the values should be as low as possible. Values below or close to 30 mN/m are considered as very good. This is especially the case if the values are reached rapidly (=at low surface ages) since it demonstrates that the surfactant is able to quickly migrate to freshly created surfaces, which is of major value in many technical applications. The results clearly demonstrate that the products of the present invention give rise to substantially lower surface tension values than the comparison products, and the low values are also reached rapidly.

### 3) Dynamic Wetting

#### Method

Measurements were carried out by depositing droplets on a hydrophobic, polymeric material (Parafilm PM-922, American Can Company) using a First Ten Angstroms FTA 200 instrument (Portsmouth, VA, USA) equipped with a video camera and an image analyzing unit. The surfactants were formulated as follows prior to measurement: Surfactant 5.0 % w/w, tetrasodium pyrophosphate (TKPP) 6.0 % w/w, sodium

metasilicate pentahydrate 4.0 % w/w and balance distilled, de-ionized water /18 MΩ/cm). The formulations were diluted 1:20 with distilled, de-ionized water before measurement. This is similar to ex. 11 in our application. Surfactant concentration however lower in this case.

## Results

**Table 3**

Product	Contact Angle (°) after		
	10 s	40 s	70 s
GT 6220-05E	49,2	45,2	42,6
GT 6220-09E	47,4	43,1	40,1
GT 6220-12E	46,4	40,3	38,7
020222HR2 (Comparison)	66,9	67,0	66,1
3xMPEG 350 (Comparison)	89,0	88,3	87,6

In this measurement a lower contact angle demonstrates better wetting of the surface. Except from giving a low equilibrium (after "long" time, e.g. one minute) contact angle it is also highly desired that the contact angle is reaching a low value already after a short time (such as 10 seconds). It is a characteristic feature of good surfactants that the contact angle is decreasing rapidly with time before it reaches a constant (equilibrium value). The contact angle for pure water on the specific surface used in this experiment is 102°.

In addition to the much higher contact angle (inferior wetting) for the products of Askew et al. the lack of surfactancy is also demonstrated by the insignificant reduction in contact angle with time.

#### 4) Wetting acc. to Draves

##### Method

The wetting capability is determined by examination of the time consumed for wetting a skein of yarn when the concentration of the solution is 1 g surfactant/liter. The skein is double-folded and equipped with a hook and a 40 g sinker of lead. Time from immersion until the lower part of the hook is on level with the upper part of the sinker is measured.

Ref. ASTM 02281

##### Results

Table 4

Product	Wetting time (s)
GT 6220-05E	41
GT 6220-09E	35
GT 6220-12E	55
020222HR2 (Comparison)	> 600
3xMPEG 350 (Comparison)	> 600

The wetting time should be as short as possible, demonstrating the ability of the surfactant to rapidly interact with the yarn surface. There exist surfactants which give extremely fast wetting (< 10 seconds) but also compounds which give wetting times < ca. 1 minute are considered to be good.

According to wetting method ASTM 02281 the products of Askew have virtually no wetting capacity, while the products according to the present invention do demonstrate significant and unexpectedly superior wetting capacity.

## 5) Emulsification

### Method

The emulsification was carried out by mixing in a 22 mL vial using an Ultra-Turrax at 8000 rpm for 60 seconds. Emulsion status was then checked with the naked eye at regular time intervals after end of emulsification. Clearly separated oil- or water phases (in mm) were noted. Rest of height was considered as emulsion phase and not noted in the table. The aqueous phase was in all cases pH 8 buffer solution (P-H TAMM Laboratorier AB, Uppsala, Sweden) in order to avoid any hydrolysis during the measurement.

### Results

Oil = decane (2g) + 1 dr Sudan Red, aq. phase = 6g, surfactant = 0.40g

Time (min)	GT 6220-08E		GT 6220-09E		GT 6220-12E		020222HR2 (Comparison)		3xMPEG 350 (Comparison)	
	Sep. oil	Sep. water	Sep. oil	Sep. water	Sep. oil	Sep. water	Sep. oil	Sep. water	Sep. oil	Sep. water
0,5	0	0	0	0	0	0	8	17	5	5
1	0	0	0	0	0	0	9	19	5	9
1,5	0	0	0	0	0	0			6	17
2	0	0	0	0	0	0			8	19
3	0	0	0	0	0	0			9	19
4	0	0	0	0	0	1				
8	1	1	0	0	0	3				
10	1	1	0	2	0	3				
15	2	2	0	2	0	6				

Grey shade = complete separation

Emulsification means mixing an aqueous and a hydrophobic liquid phase. This should normally be done as efficiently as possible (little energy required, a small amount of

surfactant required etc.), and giving an emulsion which is as long-term stable as possible.

Applicants respectfully submit that the data presented in the Declaration and discussed herein clearly demonstrate the superior emulsification properties of the compounds according to the claimed invention compared to the compounds of Askew et al. over time in that there was a complete separation of Askew et al.'s compounds after one and three minutes, respectively, while the compounds of the claimed invention show substantially better emulsification for at least as long as 15 minutes. Additionally, the compounds of the present invention were clearly superior to the compounds of Askew et al. in the following four parameters tested:

1. Static Surface Tension - wherein the products of Askew et al. have a substantially lower surface activity.

2. Dynamic Surface Tension - the surface tension of the products of the claimed invention was substantially lower than the products of Askew et al.

3. Dynamic Wetting - the products of Askew et al. had a much higher contact angle indicating inferior wetting compared to the products of the invention. Additionally, a lack of surfactancy was also demonstrated by the flat curve indicating that there was no reduction in contact angle with time.

4. Wetting according to Draves (ASTM D2281) - according to this test method the products of Askew et al. have virtually no wetting capacity, while the products according to the present invention do demonstrate significant and unexpectedly superior wetting capacity.

5. Emulsification - The data obtained clearly demonstrate the superior emulsification properties of the compounds according to the claimed invention

compared to the compounds of Askew et al. over time. More specifically, there was complete separation of Askew et al.'s compounds after 1 and three minutes, respectively, while the compounds of the claimed invention substantially better emulsification for as long as 15 minutes.

The data clearly lead one to conclude that the compounds of the present invention are unexpectedly superior surfactants compared to the compounds of Askew et al.

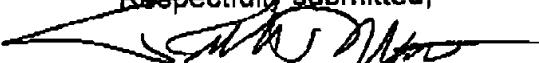
Concerning Elliott et al., this document discloses synthetic esters useful as hydraulic fluids. Elliott et al. is relied on by the examiner for allegedly showing structurally similar ortho esters to those of general formula I of the present application. Certainly the disclosed esters of Elliott are no closer to the compounds of the claimed invention than are those of Askew et al. In fact, applicants submit that the compounds of Askew et al. are clearly closer to the claimed invention than those of Elliott.

Further, it is clear from the section relied on by the examiner that Elliott, like Askew et al., contain hydrophobic parts uniformly distributed over the entire molecule, thus not resulting in clearly separated hydrophilic and (sufficiently) hydrophobic domains. As previously mentioned, a clear separation hydrophilic and (sufficiently) hydrophobic domains is known to be a requirement for a compound to orient itself at the interface between a hydrophilic (e.g. water) and hydrophobic surface and thereby induce surfactancy. In contradistinction, Elliott, like Askew et al., contain ortho esters that have three equal substituents, whereas the ortho esters according to the present invention never contain three equal substituents. Additionally, present claim 1 requires that the number of ethyleneoxy groups being at least 50% of the total number of alkyleneoxy groups in the compound of claim 1. Finally, applicants have demonstrated that the claimed compounds are unexpectedly superior to those of Askew et al. who, like Elliott, do not have a clear separation hydrophilic and (sufficiently) hydrophobic domains and which contain ortho esters that have three

equal substituents. That same data is equally pertinent to Elliott and clearly evidences that patentability of the invention thereover.

Therefore, in view of the remarks herein, the present case is believed to be in condition for allowance, which action is respectfully solicited.

Respectfully submitted,



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